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NASA TM-108449

(NASA-TM-108449) AMPOULE FAILURE SENSOR TIME RESPONSE TESTING: EXPERIMENT 1 (NASA. Marshall Space Flight Center) 15 p

N94-30199

Unclas

G3/35 0004773

AMPOULE FAILURE SENSOR TIME RESPONSE TESTING — EXPERIMENT 1

By M. L. Johnson and D. A. Watring

Astrionics Laboratory and
Space Sciences Laboratory
Science and Engineering Directorate

April 1994

NASA

National Aeronautics and
Space Administration

George C. Marshall Space Flight Center

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
April 1994

3. REPORT TYPE AND DATES COVERED
Technical Memorandum

4. TITLE AND SUBTITLE
Ampoule Failure Sensor Time Response Testing - Experiment 1

5. FUNDING NUMBERS

6. AUTHOR(S)
M. L. Johnson and D. A. Watring

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, D.C. 20546

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
NASA TM-108449

11. SUPPLEMENTARY NOTES
Prepared by the Astrionics and Space Sciences Laboratory, Science & Engineering Directorate.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified--Unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

The response time of an ampoule failure sensor exposed to a liquid or vapor gallium-arsenide (GaAs) is investigated. The experimental configuration represents the sample/ampoule cartridge assembly used in NASA's Crystal Growth Furnace (CGF). The sensor is a chemical fuse made from a metal with which the semiconductor material reacts more rapidly than it does with the containing cartridge. For the III-IV compound of GaAs, a platinum metal was chosen based on the reaction of platinum and arsenic at elevated temperatures which forms a low melting eutectic. Ampoule failure is indicated by a step change in resistance of the failure sensor on the order of megohms. The sensors will increase the safety of crystal growth experiments by providing an indication that an ampoule has failed. Experimental results indicate that the response times (after a known ampoule failure) for the 0.003 and 0.010 inch ampoule failure sensors are 2.4 and 3.6 minutes, respectively. This ampoule failure sensor will be utilized in the CGF during the second United States Microgravity Laboratory Mission (USML-2) and is the subject of a NASA patent application.

14. SUBJECT TERMS
Ampoule Failure Sensor, Response Time, Gallium-Arsenide, Microgravity

15. NUMBER OF PAGES
16

16. PRICE CODE
NTIS

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	Unlimited

NSN 7540-01-280-5500 Standard Form 298 (Rev. 2-89)

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AMPOULE FAILURE SENSOR TIME RESPONSE TESTING-EXPERIMENT 1

Introduction

Numerous ampoule failure sensor tests were successfully completed prior to this series of time response tests. These tests proved the ampoule failure sensor concept and eventual design along with their durability as they were subjected to semiconductor materials at temperatures up to 1260 °C. This test was configured to measure the response time of the ampoule failure sensor upon a known breach of an ampoule containing gallium-arsenide (GaAs) at its processing temperature. This document will discuss the experiment objectives, pre-experiment obstacles, experiment configuration, results, and conclusions.

Experiment Objectives

The primary objective of this experiment was to measure the response time of the ampoule failure sensor when exposed to GaAs at a temperature of 1260 °C. A secondary objective was to determine a wire diameter for the failure sensor that would result in the fastest response time while still enduring the high temperatures.

Ampoule Failure Sensor and Experimental Configuration

The experiment was conducted at Marshall Space Flight Center in the Hazardous Operations building, 4475, where an area was set aside for this testing. This area was approved by the Safety Office in 1990 for the purpose of developing an ampoule failure sensor. It consists of a high velocity doubled filtered fume hood. The processing furnaces were placed under the fume hood so as to contain any potentially hazardous materials that may be outgassing from the furnaces and samples under experiment. The furnaces used in this experiment were a 16 inch platinum-40% rhodium element furnace used as the primary heat source. A secondary nichrome furnace was used as a pre-heater. The pre-heating furnace sat upon the platinum furnace so as to heat the entire cartridge assembly. Figure 1 shows this experiment arrangement within the fume hood.

The sensor developed takes advantage of the high-temperature chemical reaction between the semiconductor material and the sensor material. This sensor consists of two dissimilar metals which form a closed electrical circuit. Upon ampoule failure, the sensor is immediately exposed to the molten semiconductor material and the chemical reaction causes a resistance change. In essence, the failure sensor is a chemical reactive fuse. The resistance is monitored to detect an ampoule failure. By using two dissimilar sensor materials one can measure temperature by utilizing the Seebeck effect. In this experiment the dissimilar materials were platinum and platinum-10% rhodium. Note that by no means is one limited to standard thermocouple wire materials for the sensor. A bare wire, single element, may be used without temperature measurements as long as the wire will react with the vapor or liquid semiconductor material.

The ampoule failure sensor is shown in Figure 2. It consists of a two-hole alumina protection tube with a machined flat area in which only one hole remains. The sensor wire is wrapped around this area maximizing the free surface area available for chemical reaction. The machined area also provides a larger gap between the two wires when the sensor fails. This larger gap prevents the molten semiconductor from reforming the electrical circuit once a failure has occurred. For III-V compounds, a platinum-rhodium wire combination was chosen based on the reaction of platinum and arsenic at elevated temperatures which forms a low melting eutectic. Since the diameter of the wire has primary influence on the reaction time, a 0.003 inch diameter wire was chosen for the basic design. To quantify the effects of wire size, this experiment used wire diameters of 0.003 inch and 0.010 inch for the failure sensors.

Figure 3 shows the ampoule design that was derived through experimentation. In order to know the exact time of ampoule failure, an ampoule was designed with a thin, angled fused silica tip, which included a flaw. This fused silica tip was attached at the base of the ampoule. When the ampoule is dropped, the tip breaks, resulting in the semiconductor material escaping from the ampoule.

This ampoule was then placed in a 23 inch long alumina cartridge which simulated the Crystal Growth Furnace (CGF) cartridge. The ampoule was suspended in the cartridge until the processing temperatures were achieved at which time the ampoule was released. Three ampoule failure sensors and two thermocouples were potted in an end cap and also placed in the cartridge. Two of the failure sensors were made using 0.003 inch diameter platinum wire and the other was made with 0.010 inch diameter wire. The sensors were at varying lengths in the cartridge in order to study location effects on reaction time. Two thermocouples, 0.015 inch, type S, were located at equal distances from the end cap, above the failure sensors. Additional thermocouples were placed outside the cartridge and utilized for furnace control. Figure 4 shows the relative positions of the failure sensors and thermocouples with respect to the ampoule.

The primary furnace was heated to 1260 °C. When the sample processing temperatures were achieved, the ampoule was released and allowed to fall into the 1260 °C region.

Results

During transient power up the secondary furnace resistance heater failed resulting in the secondary furnace cooling down. This was not a critical factor since the primary heater's thermal mass was sufficient enough to negate the cooling effects of the secondary heater. One of the 0.003-inch sensors also failed during transient power up. This was due to stycast potting contamination during the sensor assembly. This observation led to modifying the Ampoule Failure Sensor design to use a larger wire diameter of 0.005-inch instead of the 0.003-inch. This change increases the failure sensor's endurance to high temperature with only a minimal increase to their reaction time.

The response of the two type S thermocouples is shown in Figure 5. The temperature measured by the thermocouples experiences apparent temperature fluctuations on the order of thousands of degrees centigrade. These temperature fluctuations, observed eight minutes after ampoule failure, have the typical signature observed of a thermocouple failure due to other causes such as signal processing errors, twisted leads, grounded bead, electromagnetic interference, and/or stray voltages. These data clearly show why one cannot detect an ampoule failure based on the indicated temperature of a thermocouple.

Figures 6 and 7 show the response of the 0.003 and 0.010 inch ampoule failure sensors, respectively. The resistance on sensor MSFC-11.5 experiences a step change at an elapsed time of 149 minutes. This premature failure was due to contamination of the sensor during assembly of the test as mentioned earlier. The resistance on sensor MSFC-16 showed a step change in resistance at a time of 206.8 minutes which is 2.4 minutes after ampoule failure. For the 0.010 inch sensor, the step change occurred 3.6 minutes after ampoule failure. This clearly reveals that the key failure indication is resistance change on the order of megohms.

Conclusion

An ampoule failure sensor has been demonstrated that is capable of detecting vapor GaAs within a typical processing cartridge used in the Crystal Growth Furnace. The critical measurement is the resistance of the failure sensor which unambiguously indicates that an ampoule failure has occurred in a manner of minutes.

These sensors will increase the safety of crystal growth experiments by providing an indication that an ampoule has failed. The sensor is most beneficial for experiments performed in confined areas with limited ventilation. To this end, the failure sensors will be used in a GaAs experiment on the second United States Microgravity Mission (USML-2) in 1995. The sensor will ultimately provide increased safety and mission data return by automatically shutting down crystal growth experiments with failed ampoules.

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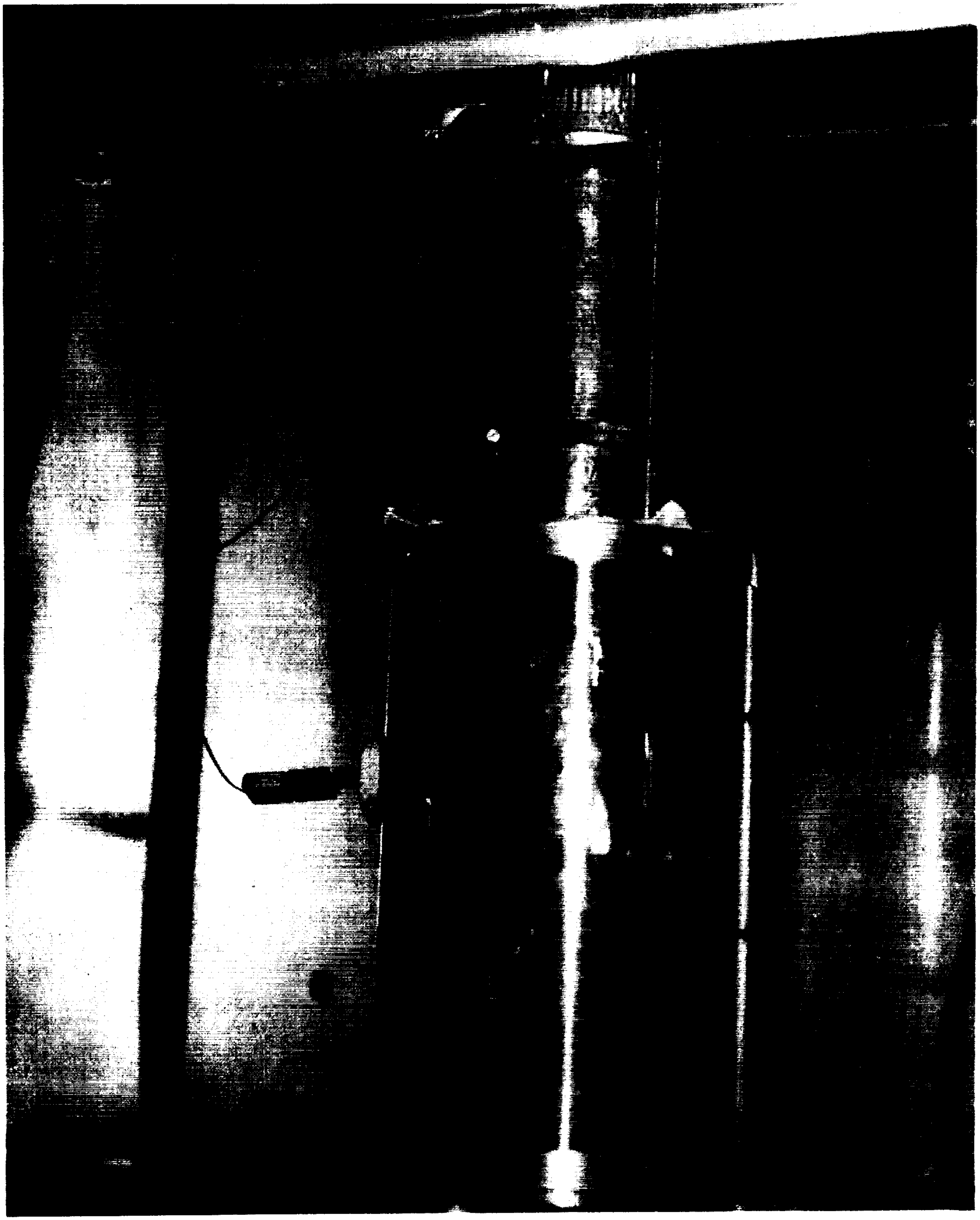

Figure 1. Furnace arrangement in fume hood.

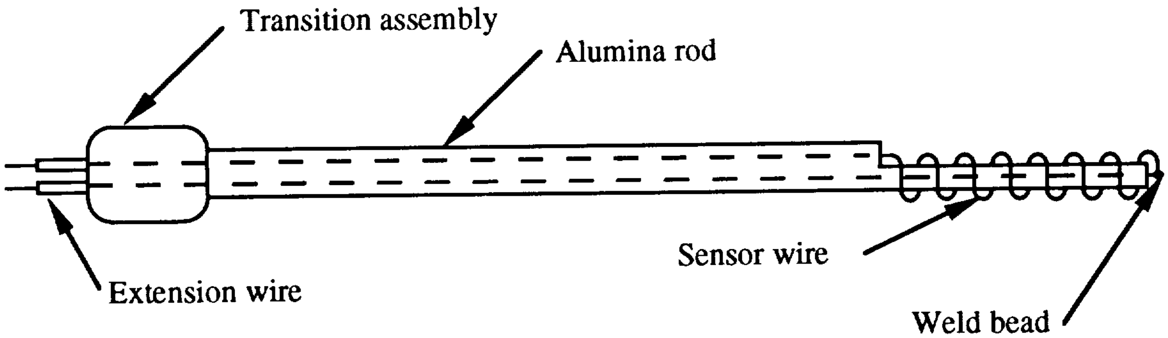

Figure 2. Schematic of ampoule failure sensor.

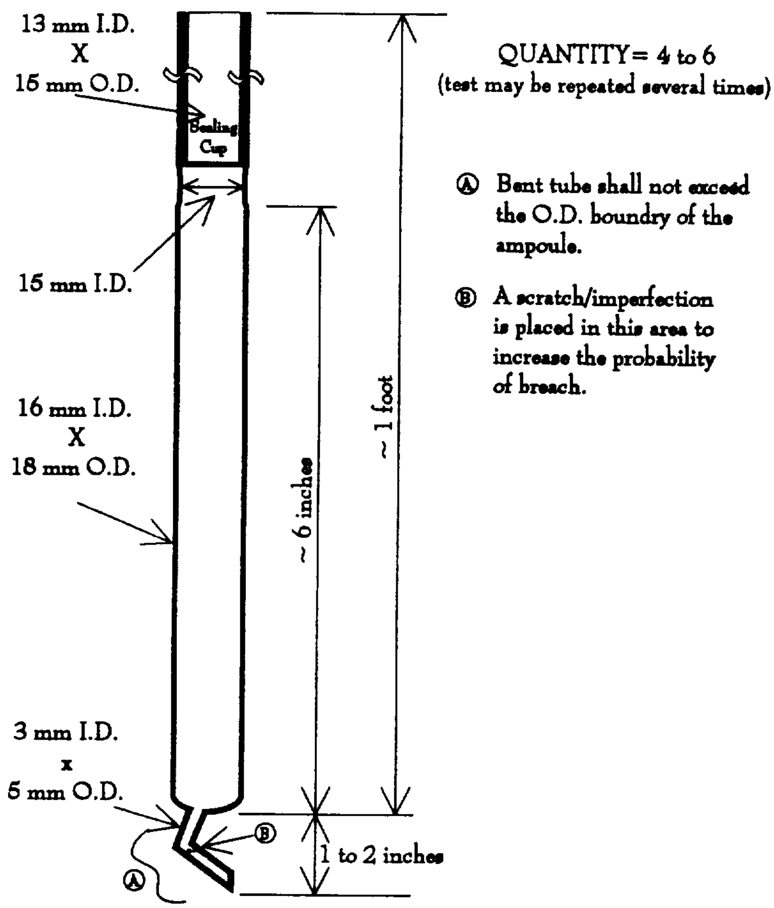

Figure 3. CGF breach test ampoule.

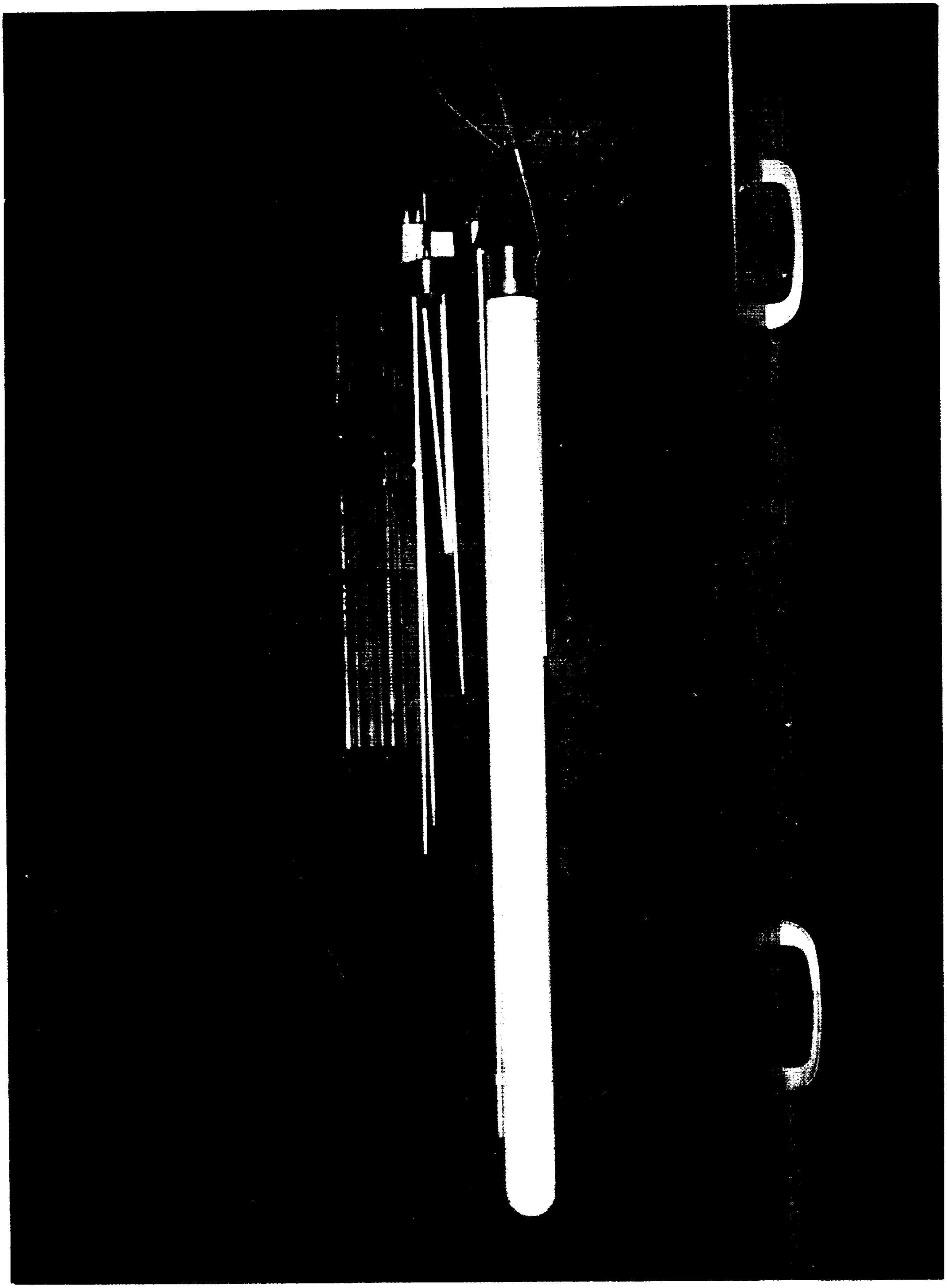

Figure 4. Experimental setup showing relative positions of failure sensors.

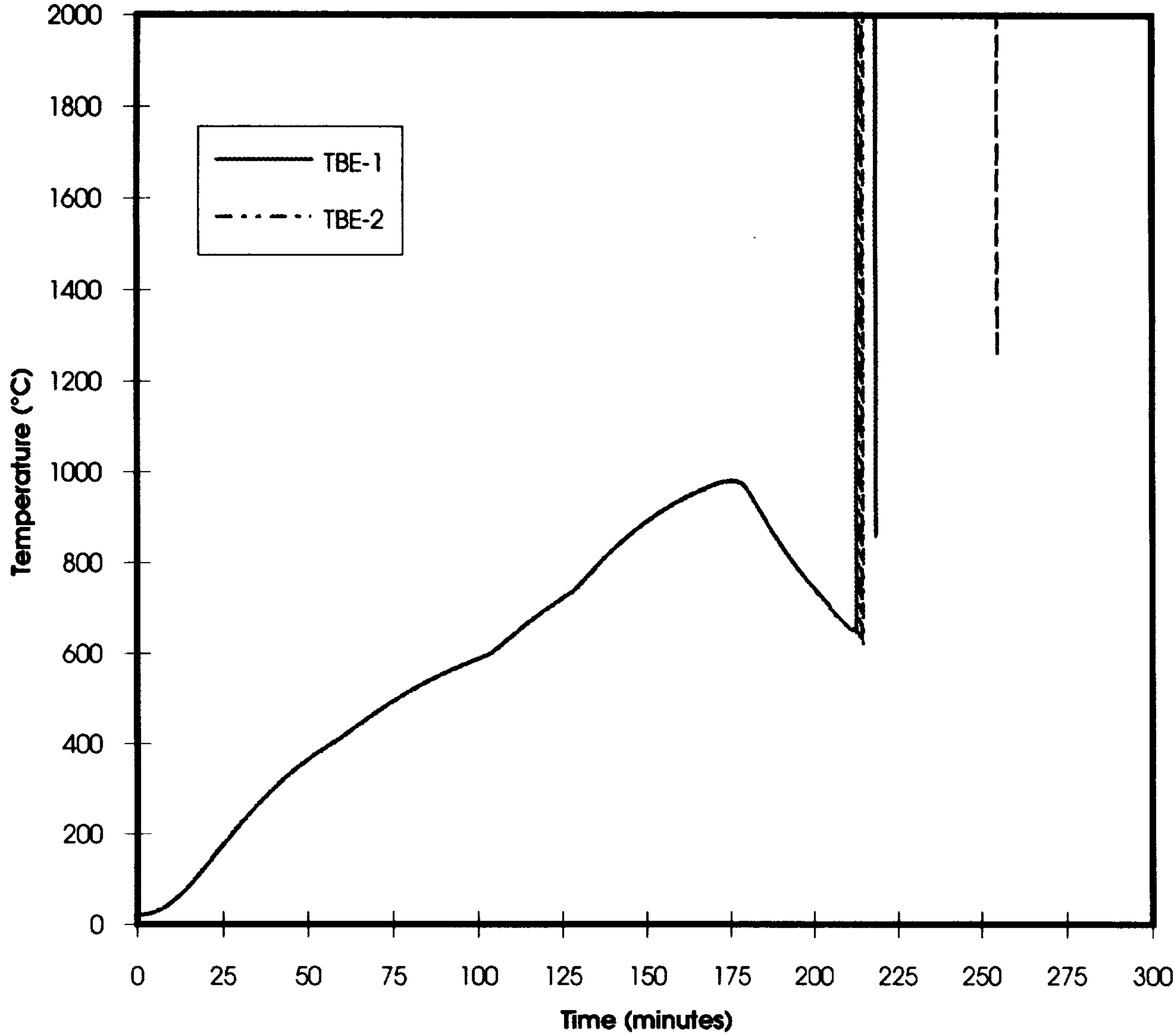

Figure 5. Temperature fluctuations for thermocouples TBE-1 and TBE-2.

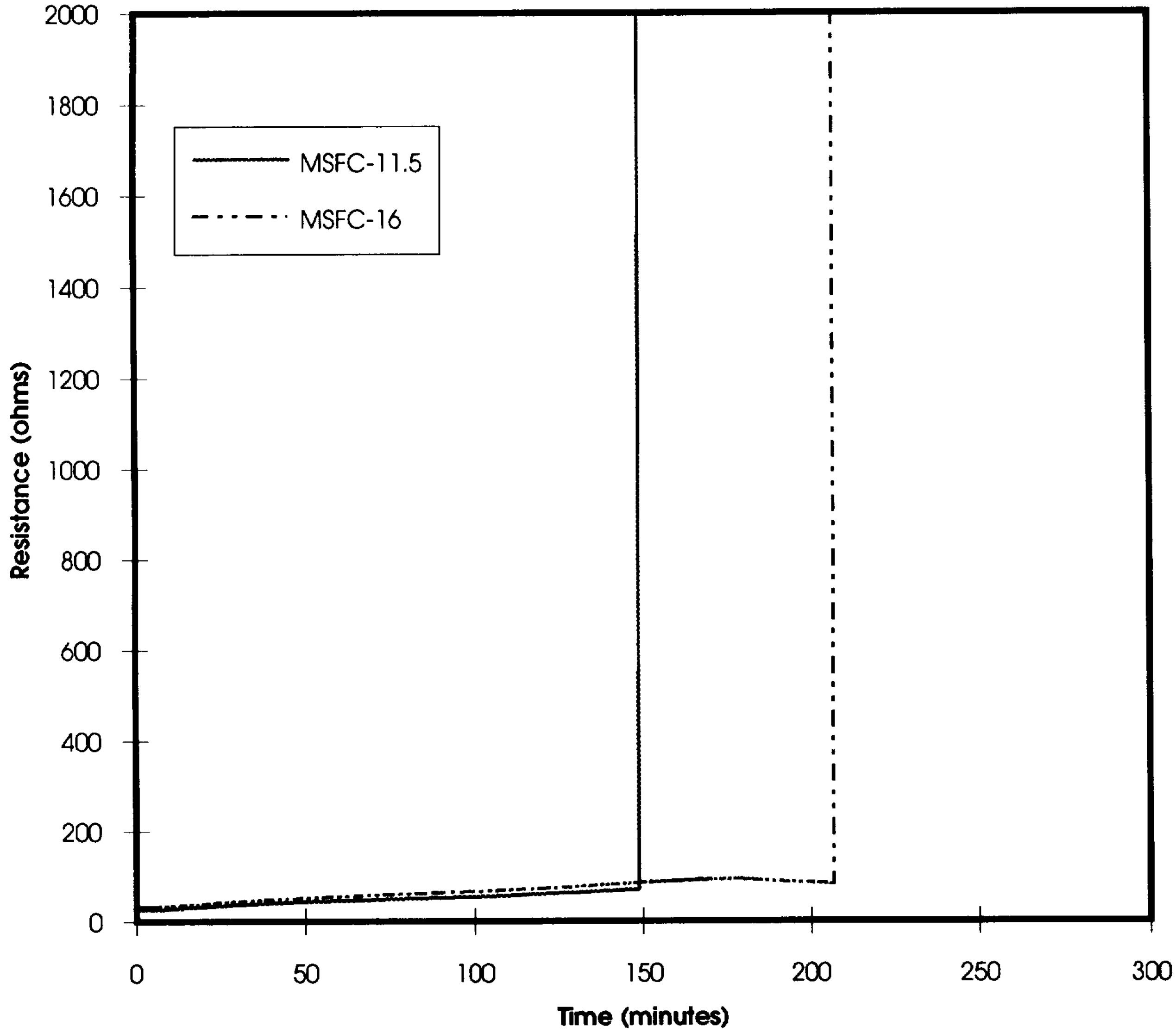

Figure 6. Resistance versus time for sensors MSFC 11.5 and MSFC 16 (0.003 inch).

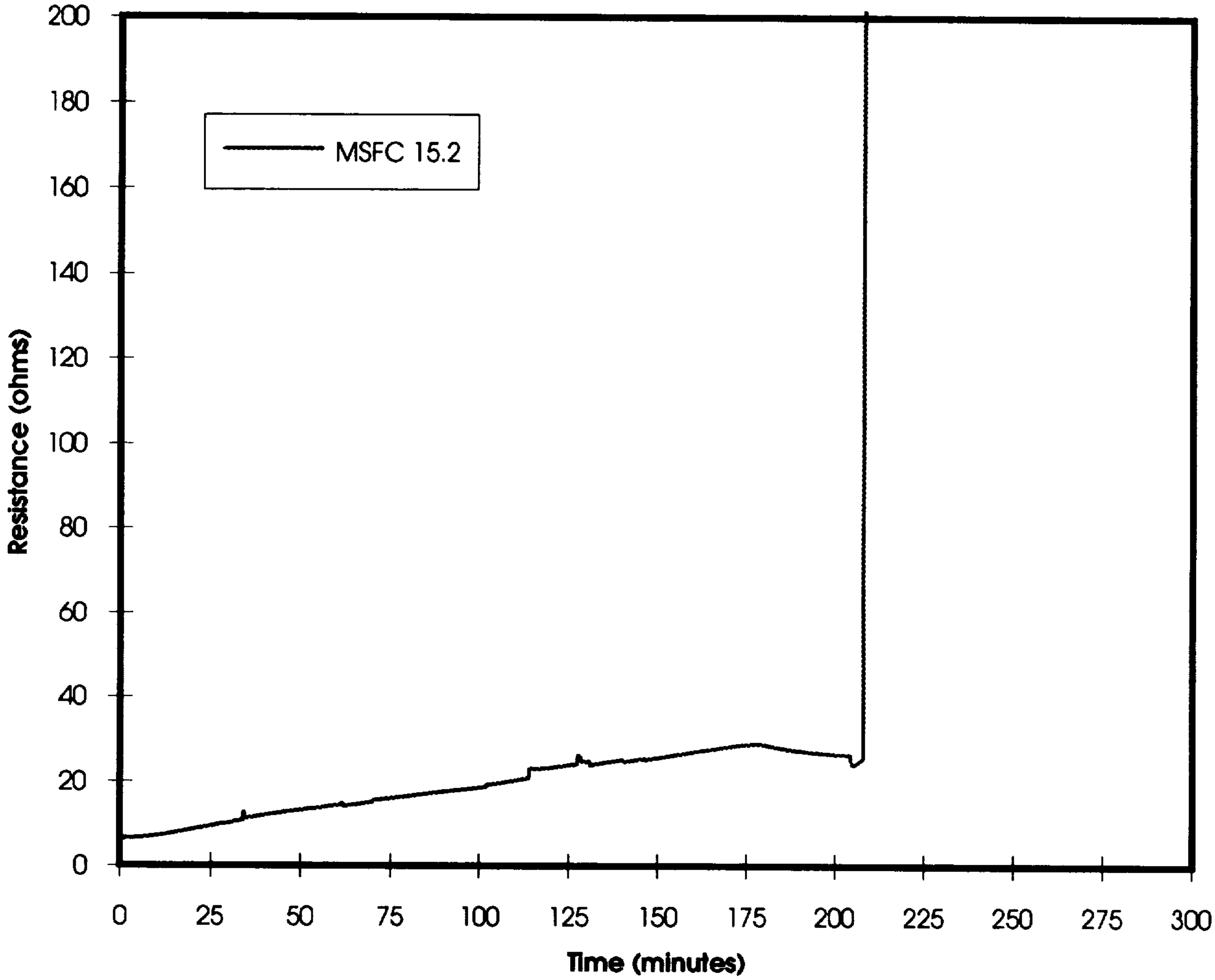

Figure 7. Resistance versus time for sensor MSFC 15.2 (0.010 inch).

APPROVAL

AMPOULE FAILURE SENSOR TIME RESPONSE TESTING - EXPERIMENT 1

By

M. L. Johnson and D. A. Watring

This report has been reviewed for technical accuracy and contains no information concerning national security or nuclear energy activities or programs. The report, in its entirety, is unclassified.

Gregory S. Wilson
Director, Space Sciences Laboratory

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